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(54) Fabric treatment composition.

(57) The siloxanes are incorporated at an amount of 0.1% to 50% by weight of said softening clay and the flocculating agents are incorporated at an amount of 0.005% to 20% by weight of said softening clay. Softening through the wash detergent compositions containing from 1% to 50% by weight of said detergent compositions of the fabric treatment compositions are also disclosed.

EP 0 483 411 A1

Technical Field

The present invention relates to fabric treatment compositions. In particular it relates to fabric treatment compositions comprising softening clays and softening polysiloxanes and flocculating agents.

Background of the Invention

The use of softening clays in fabric treatment compositions is well known in the art and has found wide commercial application. Clays have been used in particular because their application and the softening they impart on fabrics is different from that of cationic softeners. Examples and description of clay applications can be found in a large number of publications, for example U.S. 3,966,629 or U.K. 1,400,898 describe different kinds of clays and modes of application of them in fabric treatment compositions.

Combination of clay with softening amines has also been disclosed to provide softening enhancement. Further DE-3 833 648 describes improved agglomeration of detergents containing softening clay. Further a humectant is provided in EP-A-313 146 facilitating improved wetability of the clay.

Other agents which have been known for fabric treatment benefits are substituted polysiloxanes. They are disclosed for use at low levels for example in EP-A-150 867 or EP-A-150 872. Relatively high concentrations of substituted polysiloxanes are not uncommon to the textile industry and have been disclosed for example in EP-A-58 493 or U.S. 4,247,592.

In EP-A-354 856 the combination of siloxanes and cationic quaternary ammonium is disclosed.

EP-A-381 487 discloses liquid detergent compositions containing a clay component which is pretreated with siloxane to supply phase and viscosity stability while not deteriorating the cleaning performance.

It has now been found that the fabric treatment performance of substituted polysiloxanes, softening clay and a clay flocculating agent is substantially higher than was to be expected by simple additive performance. Particularly when providing intimate mixtures of softening clay and substituted polysiloxanes in the presence of clay flocculating agents the softening performance rises beyond the expectable additive softening performance of the compounds provided.

It is an objective of the invention to provide fabric treatment compositions containing substituted polysiloxanes, softening clay and a clay flocculating agent, preferably as an intimate mixture. Further it is an objective to provide softening-through-the-wash detergent compositions comprising such fabric treatment compositions, preferably granular detergent compositions.

It is another aspect of the invention to have very good environmental compatibility of the softening composition in all modes of application or use.

Summary of the Invention

The present invention relates to fabric treatment compositions comprising a softening clay, a clay flocculating agent, and a substituted polysiloxane. The substituted polysiloxane is present in said compositions at 0.1% to 50% of said softening clay and the clay flocculating agent is present at 0.005% to 20% of said softening clay.

The fabric treatment composition can be applied in liquid or granular products and used as such in the wash or in the rinse cycle of the laundry. In a preferred embodiment of the present invention said fabric treatment compositions are comprised in softening-through-the-wash (STW) detergent compositions. According to the invention STW detergent compositions contain at least one surfactant and from 1% to 50%, preferably from 1% to 20%, more preferably from 5% to 15%, of said fabric treatment compositions.

Even more preferred are particles comprising coagglomerated softening clay and substituted polysiloxanes. Together with clay flocculating agents these particles can be used in granular softening compositions or preferably in granular STW detergent compositions.

Definitions

Unless stated otherwise, the following definitions will be used hereinafter :

- percentages are percent by weight
- softening refers to a range of fabric treatments other than cleaning; in particular it includes softening, anti-wrinkling, anti-static and ease of ironing treatments.
- clays are softening clays as described in more detail below
- siloxanes are substituted polysiloxanes for fabric treatment as described in more detail below.

Detailed Description of the Invention

The softening clay

One essential component of the present compositions consists of a clay.
Any clay used in the art or mixtures thereof can be used in the present invention.

Included among such clays are various heat-treated kaolins and various multi-layer smectites. As known from the art, preferred smectite clays exhibit a cation-exchange capacity of at least 50 meq per 100 grams of clay.

Further preferred are clays which have a particle size in the 5-50 micrometer range.

Additionally preferred smectite clays are hectorite clays of the general formula

$$[(Mg_{3-x}Li_x)\ Si_{4-y}Me^{III}_{y}O_{10}(OH_{2-z}F_z)]^{-(x+y)}\ \frac{(x+y)}{n}M^{n+}$$

wherein $y = 0$; or, if $y \neq 0$, Me^{III} is Al, Fe, or B; M^{n+} is a monovalent ($n = 1$) or divalent ($n = 2$) metal ion, for example selected from Na, K, Mg, Ca, Sr. The value of $(x + y)$ is the layer charge of the hectorite clay. The hectorite clays suitable for the detergent compositions of the present invention have a layer charge distribution such that at least 50% is in the range of from 0.23 to 0.31.

Preferred are hectorite clays of natural origin having a layer charge distribution such that at least 65% is in the range of from 0.23 to 0.31.

Specific non-limiting examples of fabric softening smectite clay minerals are :

Sodium Montmorillonite

Borck R
Volclay BC ®
Gelwhite GP ®
Thixo-Jel ®
Ben-A-Gel ®

Sodium Hectorite

Veegum F ®
Laponite SP ®

Sodium Saponite

Barasym NAS 100 ®

Calcium Montmorillonite

Soft Clark ®
Gelwhite L ®
Imvite K ®

Lithium Hectorite

Barasym LIH 200 ®

The clay flocculating Agent

Clay flocculating agents are not commonly used in fabric treatment compositions. On the contrary, one is inclined to use clay dispersants, which aid in removing clay stains from fabrics. Clay flocculating agents are, however, very well known in other industries like oil well drilling, and for ore flotation in metallurgy. Most of these materials are fairly long chain polymers and copolymers derived from such monomers as ethylene oxide, acrylamide, acrylic acid, dimethylamino ethyl methacrylate, vinyl alcohol, vinyl pyrrolidone, ethylene imine. Gums, like guar gum, are suitable as well.

Preferred are polymers of ethylene oxide, acryl amide, or acrylic acid. It has been found that these polymers dramatically enhance the deposition of a clay if their molecular weights (weight average) are in the range of from 100,000 to 10 million. Preferred are such polymers having a (weight average) molecular weight of from 150.000 to 5 million, more preferably from 150,000 to 800,000.

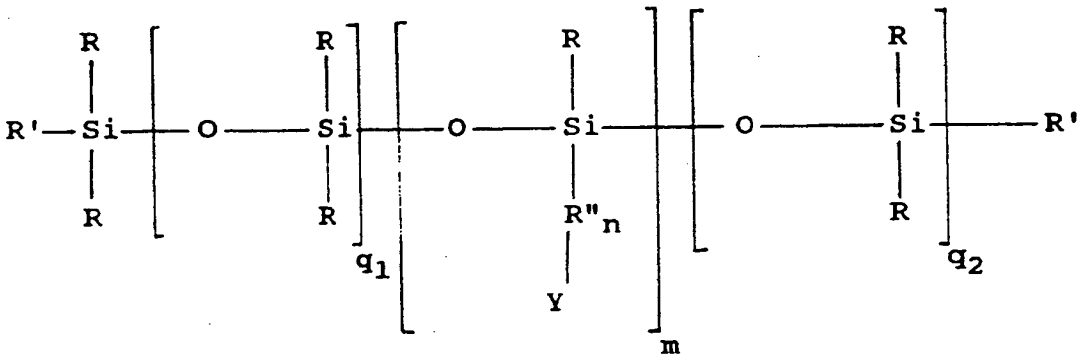
The most preferred polymer is poly-(ethylene-oxide). Molecular weight distributions can be readily determined using gel permeation chromatography, against standards of poly-(ethylene-oxide) of narrow molecular weight distributions.

The amount of clay flocculating agent, expressed as percent of the clay, ranges from 0.005% to 20%. For clay flocculating agents having a (weight average) molecular weight of less than 800,000, the preferred amount is from 2% to 20% of the clay. For (weight average) molecular weight above 800,000 the preferred amount is from 0.005% to 2% of the clay.

The subsituted polysiloxane

The amount of siloxane ranges from 0.1% to 50% by weight of the clay, preferably from 0.1% to 20%, most preferably from 1.0% to 10%.

The siloxanes useful in the present invention can be described as softening, straight or branched, organo-functional polydi-C_{1-4}-alkyl siloxane having the general formula :



wherein

R is C_{1-4}-alkyl;

R' is R or a polyether of $(C_{2-3}\text{-oxides})_{1-50}$, with a capping group of H or R;

R" is branched or straight C_{1-4}-alkyl;

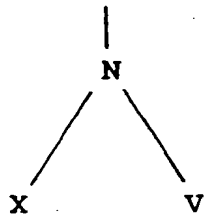
q_1 and q_2 are integers;

m and $(q_1 + q_2)$ are integers from 4 to 1700;

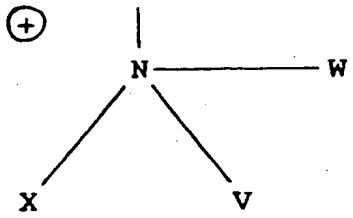
n is an integer from 0 to 6;

Y is a polyether of $(C_{2-3}\text{-oxides})_k$, where k has an average value from 7 to 100, with a capping group of H or C_{1-4}-alkyl;

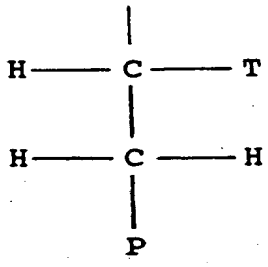
or Y is :



whereby X and V are selected from -H;
-C_{1-30}-alkyl, -C-aryl;
-C_{5-6}-cycloalkyl; -C_{1-6}-NH_2;
-COR; with the proviso that the nitrogen can be quaternized such as to represent :



whereby W can be selected from X and V.
or Y is



whereby T and P are selected from -H, -COOH, -CO-O-C_{1-2}-alkyl, or epoxy-C_{5-6}-cycloalkyl

Preferred siloxanes of said general formula are characterized by
$q_1 + q_2$ being an integer from 50 to 1500 and
m being an integer from 4 to 100.

The most preferred siloxanes of said general formula are characterized by either of the following

- R, R' is methyl and R'' is propyl and ($q_1 + q_2$) is 329 and m is 21 and n is 1 and y is a polyether consisting of 12 ethyl oxides and an acetic acid capping group or
- R, R' is methyl and R'' is propyl and ($q_1 + q_2$) is 485 and m is 15 and n is 1 and y is a polyether consisting of 12 ethyl oxides and acetic acid capping group or
- R, R' is methyl and R'' is methyl-2-propyl ($q_1 + q_2$) is 1470 and m is 30 and n is 1 and y is an -(amino ethyl)amine

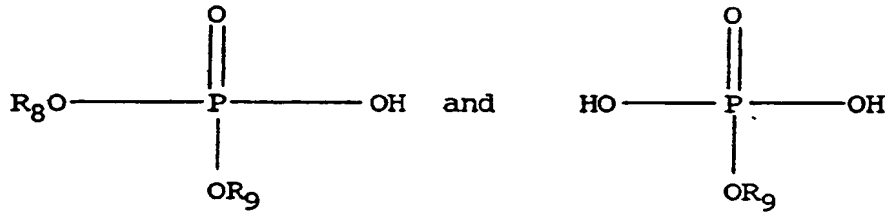
Optional softening ingredients

The fabric treatment compositions herein can comprise in addition to the essential compounds other useful compounds known in the art. These compounds can be present, depending on application and preparation needs or other desires, in an added amount of up to 85% of the fabric treatment composition.

Suitable examples among these other softening compounds include the following softening amines of the formula $R_1R_2R_3N$, wherein R_1 is C_6 to C_{20} hydrocarbyl, R_2 is C_1 to C_{20} hydrocarbyl, and R_3 is C_1 to C_{10} hydrocarbyl or hydrogen. A preferred amine of this type is ditallowmethylamine.

Preferably, the softening amine is present as a complex with a fatty acid of the formula RCOOH, wherein R is a C_9 to C_{20} alkyl or alkenyl. It is desirable that the softening amine/fatty acid complex be present in the form of microfine particles, having a particle size in the range from 0.1 to 20 micrometers. These softening amine/fatty acid complexes are disclosed more fully in EP-A-133 804. Preferred are fabric treatment compositions that contain from 1% to 10% of the amine.

Suitable are also complexes of the above described softening amine and phosphate esters of the formula



wherein R_8 and R_9 are C_1-C_{20} alkyl, or ethoxylated alkyl groups of the general formula alkyl-$(OCH_2CH_2)_y$, wherein the alkyl substituent is C_1-C_{20}, preferably C_8-C_{16}, and y is an integer of 1 to 15, preferably 2-10, most preferably 2-5. Softening amine/phosphate ester complexes of this type are more fully disclosed in EP-A-168 889.

Suitable optional softening ingredients are also the softening amines disclosed in GB 2 173 827, in particular the substituted cyclic amines. Suitable are imidazolines of the general formula

1-(C_{12-22}-alkyl)-amide-(C_{1-4}-alkyl)-2-(C_{12-22}-alkyl) imidazoline.

A preferred cyclic amine is 1-tallowamidoethyl-2-tallowimidazoline. Preferred fabric treatment compositions contain from 1% to 10% of the substituted cyclic amine.

Further examples of optional ingredients include the softening amides of the formula $R_{10}R_{11}NCOR_{12}$, wherein R_{10} and R_{11} are independently selected from C_1-C_{22} alkyl, alkenyl, hydroxy-alkyl, aryl, and alkyl-aryl groups; R_{12} is hydrogen, or a C_1-C_{22} alkyl or alkenyl, an aryl or alkyl-aryl group. Preferred examples of these softening amides are ditallow-acetamide and ditallow-benzamide. Good results are obtained when the softening amides are present in the composition in the form of a composite with a fatty acid or with a phosphate ester, as described hereinbefore for the softening amines.

The softening amides are preferably present in the fabric treatment softening composition at 1%-10%.

Moreover, the fabric treatment compositions herein can contain, in addition to ingredients already mentioned, various other optional ingredients typically used in commercial products to provide aesthetic or additional product performance benefits. Typical ingredients include pH regulants, humectants, which are more fully disclosed in EP-313 146, silicones, perfumes, dyes, hydrotropes and gel-control agents, freeze-thaw stabilizers, bactericides or preservatives. Preferred fabric treatment compositions contain from 0.05% to 30% of these ingredients.

Industrial Applications

The fabric treatment compositions of the present invention can be provided in liquid or granular form. It can be applied in the rinse cycle or, together with a detergent composition, in the wash. In a preferred embodiment said fabric treatment compositions are comprised in softening-through-the-wash (STW) detergent compositions.

Further in a preferred embodiment said fabric treatment compositions are particulate agglomerates, and in a more preferred embodiment said agglomerates are integrated in particulate detergent compositions to form granular STW detergents.

Liquid and Granular Application

The fabric treatment compositions of the present invention can be provided in liquid form as an aqueous dispersion. If provided as an aqueous dispersion, the fabric treatment composition preferably further comprises an antisettling agent. According to the invention, the aqueous dispersion comprises clay of the fabric treatment composition from 0.5% to 30% of the aqueous dispersion and siloxane and clay flocculating agent in according amounts.

A suitable antisettling agent must provide a fully activated support matrix to suspend particles within the liquid compositions.

Particles in this sense are granules or droplets of suspendable size for the desired properties of the liquid composition. Usually said particle size will be less than 200 micrometers. The individual particles can comprise one or more of the essential or optional compounds of the fabric treatment compositions.

Finally, an acceptable antisettling agent must not adversely effect the viscosity, elasticity or aesthetics of the product.

These antisettling agents, or mixtures thereof, are used in the compositions of the present invention at levels of from 0.25% to 5%.

Organophillic quaternized ammonium-clay compounds for example of the Bentone ^{R} family of clays and also fumed silicas are examples of antisettling agents suitable for use in the present invention. Bentone ^{R} rheological additives are described as the products of a clay which contains a negative layer-lattice and an organic compound which contains a cation and at least one alkyl group containing at least 10 carbon atoms. Bentones ^{R} have the property of swelling in certain organic liquids. Organophillic quaternized ammonium-clay compounds are preferred antisettling agents as described in U.S. patent 4,287,086.

Fumed silicas also provide excellent antisettling characteristics to the compositions of the present invention. Fumed silicas are generally defined as a colloidal form of silica made by combustion of silicon tetrachloride in a hydrogen-oxygen furnace. Fumed silicas are normally used as thickener, thixotropic and reinforcing agents in inks, resins, rubber, paints and cosmetics. CAB-O-SIL^{R} fumed silicas are suitable antisettling agents for use in this invention.

Other antisettling agents are cellulosic suspending agents. For example carboxy-alkyl-celluloses, preferably carboxy-methyl-cellulose, are excellent suspending agents. Furthermore other suspending agents known in the art can be applied.

Mixtures of Bentone ^{R} clays, fumed silicas or cellulosic suspending agents are also suitable antisettling agents.

The rheological characteristics of the resulting liquid compositions are very important to a commercially acceptable product. A liquid which can be described as stringy (i.e., elastic), thick or lumpy is undesirable. The antisettling agents described above avoid these undesirable rheological properties while maintaining a pourable, homogeneous product with good consumer appeal. A viscosity in the range of from about 100 to about 1000 kg/(ms) is desirable.

It is also desirable for the liquid composition to exhibit plastic rheology. Materials that exhibit plastic flow characteristics will flow only after an applied shearing stress exceeds a critical minimum value.

Fabric treatment compositions of the present invention can also be provided in granular form as particulate compositions. According to the invention particulate compositions comprise clay of the fabric treatment composition from at least 15%, preferably above 50%, of the particulate compositions and siloxane and clay flocculating agent in according amounts.

To provide particulate compositions, the compounds of said fabric treatment composition are preferably agglomerated. The typical agglomerate size useful in the present invention is from 0.2 to 1.2 millimeter on average, with individual agglomerates ranging from 0.05 mm to 2.5 mm.

According to a preferred execution of the invention the clays, siloxanes and clay flocculating agents are agglomerated together from a slurry.

In a further preferred embodiment the agglomerates containing clay and siloxane are made separately from the agglomerates containing the clay flocculating agent and both kinds of agglomerates are mixed according to the invention.

Agglomeration methods and equipment suitable for use herein include those methods known in the art. Agglomeration methods include usage of agglomeration aids including for example sodium carbonate, sodium sulfate, potassium carbonate, potassium sulfate, magnesium sulfate, lithium sulfate, lithium carbonate, sodium citrate, sodium sesquicarbonate and water. Non-limiting examples of the equipment suitable for agglomeration of clay from smaller particles include a Dravo^{R} pan agglomerator, Loedige^{R} agglomeration KG/Schugi Beldner^{R}-Granulator, whirling knife continuous vertical fluidized bed agglomerator. Niro^{R} Fluidized Bed agglomerator, Obrian^{R} Mixer/Agglomerator, and a Littleford^{R} mixer (Littleford Brothers, Inc., Florence, Kentucky, USA, eg. Model FM130D).

Other methods and equipment which use larger amounts of water, including the manufacture of agglomerates directly from a slurry, include a spray drying tower, and a prilling tower.

On a laboratory scale, food processors which are widely available to the general public can be used to agglomerate smaller clay particles into agglomerates in the disclosed size ranges.

Detergent Compositions

If applied together with detergent compositions, preferably as integral part of STW-detergent compositions the fabric treatment composition is present from 1% to 50%, preferably from 1% to 20% and most preferably from 5% to 15% of the total composition.

STW-detergent compositions of the present invention can be provided in liquid or granular form. To provide liquid or granular STW detergent compositions the detergent compounds can be either prepared and then mixed with the aqueous suspension or the granular agglomerates of the fabric treatment composition or the detergent compound and the fabric treatment composition compound can be prepared together in an integral process.

STW-detergent compositions and preferably granular SWT-detergent compositions of the present invention are characterized by comprising at least one surfactant in addition to said fabric treatment compositions. They can further contain other usual detergent compounds, in quantities common in the art.

Detersive surfactants particularly useful herein include well-known synthetic anionic, nonionic, amphoteric and zwitterionic surfactants. Typical of these are the alkyl benzene sulfonates, alkyl- and alkylether sulfates, paraffin sulfonates, olefin sulfonates, alkoxylated (especially ethoxylated) alcohols and alkyl phenols, amine oxides, alpha-sulfonates of fatty acids and of fatty acid esters, and the like, which are well-known from the detergency art. In general, such detersive surfactants contain an alkyl group in the C_9-C_{18} range; the anionic detersive surfactants can be used in the form of their sodium, potassium or triethanolammonium salts; the nonionics generally contain from about 5 to about 17 ethylene oxide groups. U.S. patent 3 995 669 contains detailed listings of such typical detersive surfactants. C_{11}-C_{16} alkyl benzene sulfonates, C_{12}-C_{18} paraffin-sulfonates and alkyl phenols are especially preferred in the compositions of the present type.

Also useful herein as the surfactant are the water-soluble soaps, e.g. the common sodium and potassium coconut or tallow soaps well-known in the art.

The detergent composition can comprise as little as 1% surfactant compositions but preferably it will contain 5% to 50%, more preferably 10% to 30% surfactants. Mixtures of the ethoxylated nonionics with anionics such as the alkyl benzene sulfonates, alkyl sulfates and paraffin sulfonates are preferred for through-the-wash cleansing of a broad spectrum of soils and stains from fabrics.

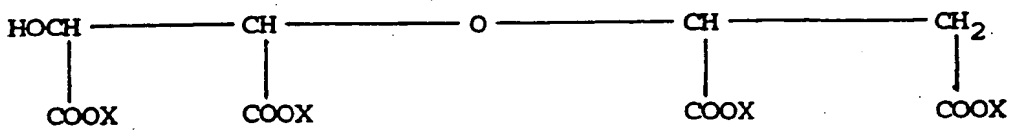
Detersive Adjuncts

The detergent composition useful in the present invention can contain other ingredients which aid in their cleaning performance. For example, it is highly preferred that detergent compositions contain a detergent builder and/or metal ion sequestrant or chelant. Compounds classifiable and well-known in the art as detergent builders include the nitrilotriacetates, polycarboxylates, citrates, carbonates, zeolites, water-soluble phosphates such as tri-polyphosphate and sodium ortho- and pyro-phosphates, silicates, and mixtures thereof. Metal ion sequestrants include all of the above, plus materials like ethylenediaminetetraacetate, the amino-polyphosphonates (DEQUEST ^{R}) and a wide variety of other polyfunctional organic acids and salts too numerous to mention in detail here. U.S. patent 3 579 454 discloses typical examples of the use of such materials in various cleaning compositions. In general, the

builder/sequestrant will comprise about 0.5% to 45% of the total composition. The 1-10 micron size zeolite (e.g. zeolite A) builders; disclosed in German patent 2 422 655, are especially preferred for use in low-phosphate compositions.

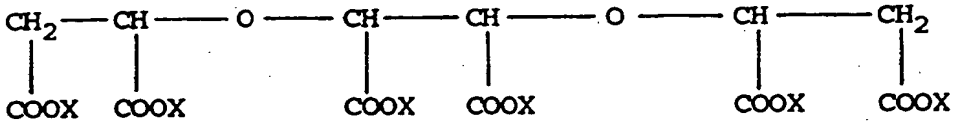
Particularly suitable phosphate-free builders are ether carboxylate mixtures comprising

a) from 1% to 99% of the builder of a tartrate monosuccinate component of the structure



wherein X is H or salt-forming cation; and

b) from 1% to 99% of the builder by weight of a tartrate discussinate component of the structure :



wherein X is H or a salt-forming cation.

Builder systems of this type are more fully disclosed in U.S. patent 4,663,071.

Typical detergent compositions useful in the present invention contain from 5% to 35% of this builder system.

The detergent compositions herein also preferably contain enzymes or enzyme mixtures to enhance their cleaning performance on a variety of soils and stains. Hydrolases and isomerases like amylase, lipase, cellulase, and protease enzymes suitable for use in detergents are well-known in the art and in commercially available liquid and granular detergents. Commercial detersive enzymes (preferably a mixture of amylase, lipase, cellulase and protease) are typically used at levels of 0.001% to 5% of said detergent compositions. Detergent cellulase enzymes, which provide both cleaning and softening benefits, particularly to cotton fabrics, are highly desirable in the compositions of this invention.

Further said detergent compositions can contain other ingredients which aid in their cleaning performance. For example, the compositions herein can advantageously contain a bleaching agent, especially a peroxyacid bleaching agent. In the context of the present invention, the term peroxyacid bleaching agent encompasses both peroxyacids per se and systems which are able to yield peroxyacids in situ.

Peroxyacids per se are meant to include the alkaline and alkaline-earth metal salts thereof. Peroxyacids and diperoxyacids are commonly used; examples are diperoxydodecanoic acid (DPDA) or peroxyphthalic acid.

Systems capable of delivering peracids in situ consist of a peroxygen bleaching agent and an activator thereof.

The peroxygen bleaching agents are those capable of yielding hydrogen in an aqueous solution; these compounds are well-known in the art, and include hydrogen peroxide, alkali-metal peroxides, organic peroxide bleaching agents such as urea peroxide, inorganic persalt bleaching agents such as alkali metal perborates, percarbonates, perphosphates and persilicates.

The liberated hydrogen peroxide reacts with a bleach activator to form the peroxyacid bleach. Classes of bleach activators include esters, imides, imidazoles, oximes, and carbonates. In these classes, preferred materials include methyl o-acetoxy benzoates; sodium-p-acetoxy benzene sulfonates such as sodium-4-nonanoxyloxybenezene sulfonate, and sodium-4-decanoyloxybenzenesulfonate : biophenol A diacetate; tetra acetyl ethylene diamine; tetra acetyl hexamethylene diamine; tetra acetyl methylene diamine.

Other highly preferred peroxygen bleach activators which are disclosed in U.S. patents 4.483.778 and 4.539.130, are alpha-substituted alkyl or alkenyl esters, such as sodium-4(2-chlorooactanoyloxy)benzene sulfonate, sodium 4-(3,5,5-trimethyl hexanoyloxy)benzene sulfonate. Suitable peroxyacids are also perox-

ygen bleach activators such as described in EP-A-116 571, i.e., compounds of the general type RXAOOH and RXAL, wherein R is a hydroxcarbyl group, X is a hetero-atom, A is a carbonyl bridging group and L is a leaving group, especially oxybenzenesulfonate.

Also polymeric soil release agents are useful in the detergent compositions of the present invention. They include cellulosic derivatives such as hydroxyether cellulosic polymers, copolymeric blocks of ethylene terphthalate and polyethylene oxide or polypropylene oxide terephthalate, cationic guar gums, and the like.

The cellulosic derivatives that are functional as soil release agents are commercially available and include hydroxyethers of cellulose such as Methocel^{R} (Dow) and cationic cellulose ether derivatives such as Polymer JR-124^{R}, JR-400^{R}, and JR-30M^{R} (Union Carbide).

Other effective soil release agents are cationic guar gums such as Jaguar Plau^{R} (Stein Hall) and Gendrive 458^{R} (General Mills).

Preferred cellulosic soil release agents for use herein have a viscosity in aqueous solution at 20°C of 15 to 75,000 kg/(ms) and are selected from the group consisting of methyl cellulose; hydroxypropyl methylcellulose, hydroxybutyl methylcellulose, or mixtures thereof.

A more preferred soil release agent is a copolymer having random blocks of ethylene terphthalate and polyethylene oxide (PEO) terephthalate. More specifically, these polymers are comprised of repeating units of ethylene terephthalte and PEO terephthalate in a mole ratio of ethylene terephthalate units of from about 25:75 to about 35:65, said PEO terephthalate units containing polyethylene oxide having molecular weights of from 300 to 2000. The molecular weight of this polymeric soil release agent is in the range of from 25,000 to 55,000. U.S. patents 3,959,230 and 3,893,929 disclose similar copolymers in a large variety. It has been found previously that these polymeric soil release agents provide a more uniform distribution over a range of fabrics and can therefore yield improved fabric care qualities.

Another preferred polymeric soil release agent is a crystallizable polyester with repeat units of ethylene terephthalate units containing about 10-15% by weight of ethylene terephthalate units together with about 80% to about 90% by weight of polyoxyethylene terephthalate units, derived from a polyoxyethylene glycol of average molecular weight 300-5,000, and the mole ratio of ethylene terephthalate units to polyoxyethylene terephthalate units in the crystallizable polymeric compound is between 2:1 and 6:1. Examples of this type of polymer include the commercially available material Zelcon^{R} 5126 (from Dupont) and Milease^{R} T (from ICI).

Preferred soil release polymers and methods for their preparation are described in EP-A-185,417.

If utilized, these soil release agents will generally comprise from 0.05% to 5% of the detergent compositions.

EXAMPLES

The following examples illustrate preferred executions of this invention, and facilitate its understanding.

Abbreviations for individual ingredients in the examples have the following meaning :

LAS :	Sodium salt of linear dodecyl benzene sulfonate
TAS :	Sodium salt of tallow alcohol sulfate
AOS :	Sodium salt of alpha-olefin (C12-18) sulfonate
TAE-11 :	Tallow alcohol ethoxylated with about 11 moles of ethylene oxide
FA25E7 :	Fatty alcohol (C12-15) ethoxylated with 7 moles of ethylene oxide
DTMA :	Ditallow methyl amine
CFA :	Coconut fatty acid
HFA :	Hydrogenated C16-22 fatty acid
Zeolite A :	Sodium salt of fully hydrated zeolite 4A (average particle size between 2-6 microns)
NTA :	Sodium salt of nitrilotriacetate
Copolymer :	AA^{30}/MA^{70} = copolymer of acrylic acid 30 mole-% and maleic acid 70 mole-%-50-70 Mmole weight
CMC :	Sodium salt of carboxymethylcellulose
EDTA :	Sodium salt of ethylene diamine tetra-acetate

Example I

For compositions A,B,A',B' the following was prepared : 500g of smectite clay having a cation exchange capacity of 70-80meq/100g was mixed with 50g polysiloxane (10% of clay). The polysiloxane was of the general formula with R being methyl, R' being a strait propyl, q being 329, m being 21, n being 1 and y

being a polyether of 12 ethyl oxides capped with acetic acid. Both compounds were intimately mixed and agglomerated by using a Braun^{R} multipractic-electronic-de-luxe mixer. The agglomeration aid was water and the agglomerates were sieved to a particle size from 0.15 to 0.85 mm. A reference without the siloxane was also prepared in the same manner.

Composition A was prepared by dry mixing of the clay/siloxane granules with the detergent granules of composition I, according to Table I, such that the resulting softening-through-the-wash detergent contained 10.5% of the smectite clay. As a reference composition B was prepared by dry mixing the clay granules with the same detergent granules of composition 1, Table 1, such that the resulting STW detergent also contained 10.5% of the smectite clay. Further compositions A',B' were prepared in the same way as compositions A,B respectively but using detergent composition II of Table I which contains clay flocculating agent.

To facilitate a softness comparison of A,B,A',B' the following test procedure was used :
3.5 kg of clean fabric laundry loads are washed in an automatic drum washing machine Miele^{R} 423 at 60 °C for 1.5 hours. The hardness of the water was 3.0 mmol of Ca^{2+} and Mg^{2+} per liter and the composition concentration was 1% in the wash liquid. For softness evaluation swatches of terry towel softness tracers were added. The softness tracers were line dried prior to assessment of softness. Comparative softness assessment was done by expert judges using a scale of 0 to 4 panel-score-units (PSU). In this scale 0 is given for no difference and 4 is given for maximum difference. Softness was assessed after one and after four wash cycles.

Softness evaluation results of compositions A,B,A',B'

Result		
comparison of composition	one wash cycle	four wash cycles
1. A to B	- 0.1 PSU	0.0 PSU
2. A' to B'	0.6 PSU	0.9 PSU

Taking result 1 there is no additive softening effect realized by adding siloxane to a clay-containing STW detergent. The effect of the invention is demonstrated in result 2 where the combination of clay, flocculating agent and siloxane shows surprisingly clear softening superiority over prior art composition containing clay and flocculating agent.

Example II

The same compositions a,b,a',b' respectively as in example I were prepared except for using 5g of polysiloxane, i.e. 1% of the weight of the clay. The polysiloxane used in this example II was Silwet L720 from Union Carbide.

Result		
comparison of composition	one wash cycle	four wash cycles
3. a to b	- 0.2	0.1
4. a' to b'	0.3	0.6

Examples III - XII

According to the invention other detergent compositions can be used together with the clay-siloxane agglomerates and flocculating agent. Alternative granular detergent compositions are listed in Table II.

Similar compositions in which the clay/siloxane granules are initially dry mixed with granules containing the flocculating agent have been found to be equally effective when added to granular detergent compositions.

Example XIII

Exemplifying usage of the fabric treatment composition as aqueous dispersion the following composition showed excellent softening performance when added in an appropriate amount to the last rinse of the wash :
80g of smectite clay are dispersed in 1 kg of water. 8g of siloxane as described in example I or II are added under strong agitation. The flocculating agent and this mixture are added together to the last rinse of the wash.

Example XV

A stabilized aqueous dispersion of the fabric treatment composition is prepared by using a propeller mixer at rotation of 100 to 1000 per second and combining liquid carrier, softening clay, siloxane and an antisettling agent in said propeller mixer for about 30 minutes. The resulting mixture is then passed 4 to 10 times through a colloid mill (e.g. model SD-40, distributed by Tekmor^{R} Corporation) providing a shear of 10-40x10^3 per second at a temperature of 20-40 ° C. Then the flocculating agent is added slowly using said propeller mixer for an additional 5 minutes.

The resulting mixture is stable, i.e. shows now phase separation or settling of the clay, for several weeks at ambient conditions.

Table I

Composition of granular detergents. All values in weight percent.

	Composition	
	I	II
LAS	8	8
TAS	2	2
C_{12}-C_{14} Dimethyl(hydroxyethyl)ammonium chloride	2	2
FA25E7	1	1
Zeolite A	23	23
Copolymer	5	5
Diethylene tri-amine penta (methylene phosphonic acid)	0.5	0.5
Sodium perborate (1 aq.)	12	12
Tetraacetylethylene diamine	4	4
Zn phtalocyanine sulphonate	20ppm	20ppm
Perfume	0.4	0.4
Enzyme (protease)	2	2
Montmorrillonite clay	11	11
Glycerol	0.5	0.5
Polyethylene oxide (MW 300.000)	-	0.25
Sodium carbonate	11	11
Sodium silicate	5	5
Silicone/silica suds suppressor	1	1
CMC	0.4	0.4
Optical brightener	0.2	0.2
moisture and minor	balance to 100	

Table II

INGREDIENT	COMPOSITIONS (% by weight)									
	III	IV	V	VI	VII	VIII	IX	X	XI	XII
LAS	7.0	5.0	4.0	1.0	6.5	20.1	6.7	7.0	8.0	-
TAS	-	2.0	-	-	1.0	-	-	-	2.0	2.0
C_{14-15}alkyl sulfate (Na)	-	-	-	-	-	-	6.7	-	-	-
AOS	-	-	2.0	-	-	-	-	-	-	-
Tallow-amino C_{1-4}-alkyl glucose amide	-	-	-	-	-	-	-	-	-	8.0
TAE-11	1.0	2.0	2.0	-	0.8	-	-	1.0	-	-
FA25E7	-	-	-	6.0	-	-	1.0	-	1.0	1.0
HFA	2.5	1.0	-	1.0	1.0	-	-	2.5	-	-
CFA	-	-	1.5	-	-	-	-	-	-	-
C_{12}-C_{14} Dimethyl (hydroxy-ethyl) ammonium cloride	-	1.5	-	-	1.5	-	-	-	2.0	2.0
Sodium tripolyphosphate	24.0	18.0	22.0	32.0	-	36.9	26.3	24	-	-
Zeolite A	-	-	-	-	20.0	-	-	-	23.0	23.0
Polyethylene oxide 5MM mole wt	0.05	-	-	-	0.05	0.1	0.1	-	-	-
Polyethylene oxide 0.3MM mole wt	-	-	-	0.2	-	-	-	0.2	0.25	-
Polyacrylate	-	0.2	-	-	-	-	-	-	-	-
Polyacrylamide	-	-	1.5	-	-	-	-	-	-	-
NTA	-	-	-	-	5.0	-	-	-	-	-
Sodium sulfate	12.4	17.7	15.0	21.3	12.7	28.3	13.0	12.4	-	-
Sodium carbonate	-	8.0	-	5.0	-	-	15.0	5.0	11.0	11.0

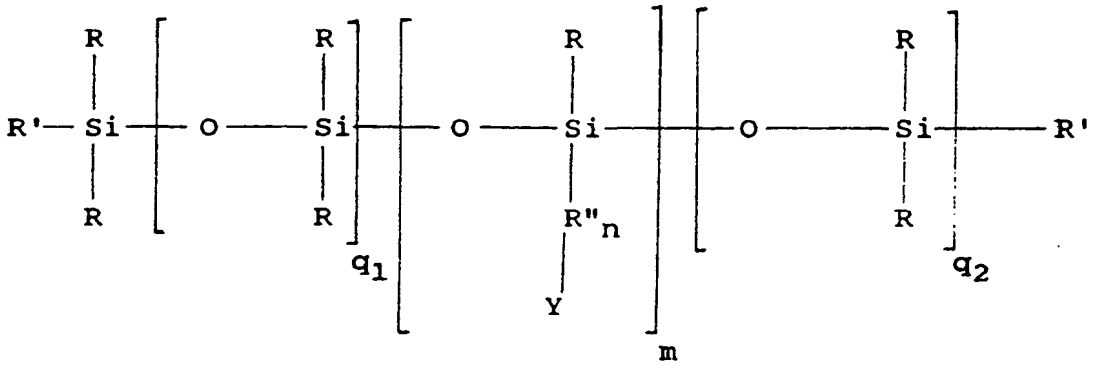
Table II - Continued

INGREDIENT	COMPOSITIONS (% by weight)									
	III	IV	V	VI	VII	VIII	IX	X	XI	XII
Sodium silicate	6.0	7.0	4.0	6.0	2.0	5.7	5.6	6.0	5.0	5.0
Sodium perborate (4aq.)	20.0	15.0	18.0	10.0	18.0	-	5.0	15.0	-	-
Sodium perborate (laq.)	-	-	-	-	-	-	-	-	12.0	12.0
CMC	0.3	0.3	0.5	0.8	0.4	-	-	0.3	0.4	0.4
Polyacrylate (MW1000-20000)	-	1.5	-	-	-	-	0.80	-	-	-
Polyacrylate (MW4000-5000)	-	-	-	-	3.0	-	-	-	-	-
Copolymer	2.0	-	1.5	2.5	-	-	-	2.0	4.0	5.0
Enzymes (protease, amylase, lipase, cellulase)	0.6	0.2	0.5	0.5	0.3	-	0.6	0.6	2.0	2.0
Optical brightener	0.2	0.2	0.3	0.3	0.25	-	0.8	0.2	0.4	0.4
Zn phthalocyanine sulphonate	30ppm	-	-	25ppm	25ppm	-	-	30ppm	20ppm	20ppm
EDTA	0.2	0.2	0.3	0.15	0.2	-	0.1	0.2	-	-
Ethylenediamine tetramethylene phosphonic acid	0.2	0.1	-	0.1	0.1	-	-	0.2	-	-
Diethylenetriamine penta (methylene phosponic acid)	-	-	-	-	-	-	-	-	0.2	0.5
Tetraacetyl ethylenediamine	1.5	-	-	-	1.5	-	-	2.0	4.0	4.0
Iso-nonanoyloxy-benzene sulfonate (Na)	-	2.0	-	-	-	-	2.0	-	-	-
Silicone/silica suds suppressor	0.2	0.15	0.15	0.25	0.2	-	0.30	0.2	1.0	1.0
Perfume	0.25	0.25	0.30	0.2	0.25	0.2	0.25	0.25	0.4	0.4
Montmorillonite clay	10.0	7.0	15.0	5.0	10.0	-	5.6	9.5	11.0	11.0
Hectorite clay	-	-	-	-	-	8.9	-	-	-	-
Moisture and minors	balance to 100									

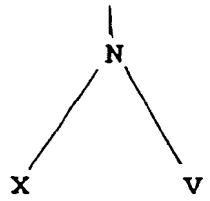
Claims

1. A fabric treatment composition comprising

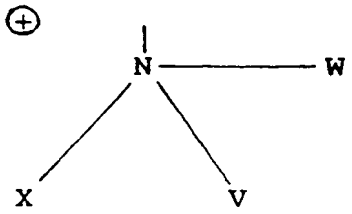
a softening clay and
- 0.005%-20% by weight of said softening clay of a clay flocculating agent and
- 0.1%-50% by weight of said softening clay of straight or branched, substituted polysiloxane of the general formula



wherein R is C_{1-4}-alkyl;
R' is R or a polyether of $(C_{2-3}\text{-oxides})_{1-50}$, with a capping group of H or R;
R'' is branched or straight C_{1-4}-alkyl;
q_1 and q_2 are integers;
m and $(q_1 + q_2)$ are integers from 4 to 1700;
n is an integer from 0 to 6;
Y is a polyether of $(C_{2-3}\text{oxides})_k$, where k has an average value from 7 to 100, with a capping group of H or C_{1-4}-alkyl;
or Y is :

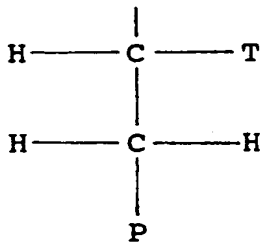


whereby X and V are selected from -H;
$-C_{1-30}$-alkyl, -C-aryl;
$-C_{5-6}$-cycloalkyl; $-C_{1-6}$-NH_2;
-COR; with the proviso that the nitrogen can be quaternized such as to represent :



whereby W can be selected from X and V.

or Y is



whereby T and P are selected from -H, -COOH, -CO-O-C_{1-2}-alkyl, or epoxy-C_{5-6}-cycloalkyl

2. A fabric treatment composition according to any of the previous claims characterized in that said softening clay is a smectite clay with a cation exchange capacity of at least 50 meq/100 gr.

3. A fabric treatment composition according to claims 1-4 characterized in that said clay flocculating agent has a weight average molecular weight from 800,000 to 5,000,000 and is present from 0.005% to 2% of said softening clay amount.

4. A fabric treatment composition according to claims 1-4 characterized in that said clay flocculating agent has a weight average molecular weight from 150,000 to 800,000 and is present from 2% to 20% of said softening clay amount.

5. A fabric treatment composition according to any of the previous claims characterized in that the substituted polysiloxane amount ranges from 0.1% to 20% of said softening clay amount.

6. A fabric treatment composition according to any of the previous claims characterized in that the substituted polysiloxane amount ranges from 1% to 10% of said softening clay amount.

7. A fabric treatment composition according to any of the previous claims characterized in that the substituted polysiloxane of the general formula of claim 1 has said integers (q_1 + q_2) from 50 to 1500 and said integer m from 4 to 100.

8. A fabric treatment composition according to any of the previous claims characterized in that the substituted polysiloxane of the general formula of claim 1 is selected from either
   - R, R' is methyl and R'' is propyl and (q_1 + q_2) is 329 and m is 21 and n is 1 and y is a polyether consisting of 12 ethyl oxides and an acetic acid capping group or
   - R, R' is methyl and R'' is propyl and (q_1 + q_2) is 485 and m is 15 and n is 1 and y is a polyether consisting of 12 ethyl oxides and acetic acid capping group or
   - R, R' is methyl and R'' is methyl-2-propyl (q_1 + q_2) is 1470 and m is 30 and n is 1 and y is an -(amino ethyl)amine.

9. A fabric treatment composition according to any of the previous claims characterized in that it further contains softening amines in an amount from 1% to 10% by weight of said fabric treatment composition.

10. A fabric treatment composition according to any of the previous claims characterized in that it further contains humectant in an amount from 1% to 10% by weight of said fabric treatment composition.

11. A fabric treatment composition according to any of the previous claims characterized in that it further contains silicone in an amount from 1% to 30% by weight of said fabric treatment composition.

12. A fabric treatment composition according to any of the previous claims characterized in that it is in the form of an aqueous dispersion and containing said softening clay in an amount from 0.5% to 30% by weight of said aqueous dispersion.

13. An aqueous dispersion according to claim 13 characterized in that it also contains an antisettling agent at an amount from 0.25% to 5% by weight of said aqueous dispersion.

14. A fabric treatment composition according to claims 1 to 11 characterized in that it is in the form of granular agglomerates having an average size range of 0.5mm to 1.2mm with individual agglomerates having a size range from 0.05mm to 2.5mm and comprising said softening clay in an amount of at least 15% by weight of said fabric treatment composition.

15. A fabric treatment composition according to claim 14 characterized in that it comprises said softening clay in an amount of more than 50% by weight of said fabric treatment compositions.

16. A laundry detergent composition comprising at least one surface active agent characterized in that it also contains from 1% to 50% by weight of said laundry detergent composition of the fabric treatment composition according to any of the previous claims.

17. A laundry detergent composition according to claim 16 characterized in that said fabric treatment composition is present in an amount from 1% to 20%, preferably 5% to 15%, by weight of said laundry detergent composition.

18. A laundry detergent composition according to claims 16 or 17 characterized in that it further comprises a detergent builder in an amount from 0.5% to 45% by weight of said laundry detergent composition.

19. A laundry detergent composition according to claims 16, 17 or 18 characterized in that it further comprizes amylase, lipase, cellulase or protease enzymes or mixtures thereof in an amount from 0.001% to 5% by weight of said laundry detergent composition.

20. A laundry detergent composition according to claims 16, 17, 18 or 19 characterized in that it further comprizes a bleaching agent or soil release agent.

21. A laundry detergent composition according to claims 16 to 20 characterized in that said laundry detergent composition is liquid.

22. A laundry detergent composition according to claims 16 to 20 characterized in that said laundry detergent composition is granular.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 20 2868

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,Y	EP-A-150867 (PROCTER & GAMBLE) * claims 1-5 * examples 1-3, 5-8 *	1-6, 9-12, 14, 16-22	C11D3/12 C11D3/37
Y	EP-A-299575 (PROCTER & GAMBLE) * claims 1-14 * examples *	1-6, 9-12, 14, 16-22	
A	EP-A-328182 (PROCTER & GAMBLE) * page 15; claims 1-9 *	1-4, 12, 13	
A	US-A-4639321 (C. BARRAT) * column 6, line 57 - column 7, line 27; claims 1-15 *	1, 2, 5, 6, 8, 12, 16-21	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	18 JUNE 1991	PFANNENSTEIN H.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
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